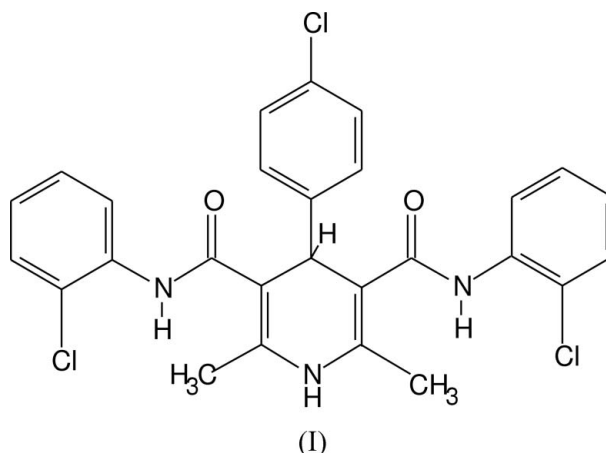


N,N'*-Bis(2-chlorophenyl)-4-(4-chlorophenyl)-2,6-dimethyl-1,4-dihydropyridine-3,5-dicarboxamide*M Mahendra,^a
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mas@physics.uni-mysore.ac.in**Key indicators**Single-crystal X-ray study
T = 295 K
Mean $\sigma(\text{C}-\text{C})$ = 0.005 Å
R factor = 0.051
wR factor = 0.181
Data-to-parameter ratio = 14.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

In the title compound, $\text{C}_{27}\text{H}_{22}\text{Cl}_3\text{N}_3\text{O}_2$, the dihydropyridine ring has a boat conformation. The molecules are linked by $\text{N}-\text{H}\cdots\text{O}$, $\text{N}-\text{H}\cdots\text{Cl}$, $\text{C}-\text{H}\cdots\text{N}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

Comment

1,4-Dihydropyridines are known for their pronounced drug activity, such as calcium channel antagonists (Janis & Triggle, 1983) and antihypertensive agents (Fossheim *et al.*, 1982). Recently, symmetrical 1,4-dihydropyridines (1,4-DHP) having 3,5-dicarbamoyl substituents (Desai *et al.*, 2001) opened up a fresh approach to the synthesis of new molecules having better pharmacokinetic properties (Triggle, 2003). Our group has synthesized and studied antitubercular activity by applying two- and three-dimensional quantitative structure–activity relationships (QSAR) to a series of 3,5-dicarbamoyl functionalities in 1,4-dihydropyridine (Desai *et al.*, 2001; Kharkar *et al.*, 2002). It was assumed that the presence of a group such as Cl or CH_3 at position 2 of the substituted phenylcarbamoyl group decreased the solvent-accessible surface area around the amide bond and hence decreased the antitubercular activity. However, when an electron-withdrawing group is present at position 2 of the phenylcarbamoyl group it results in higher activity (Kharkar *et al.*, 2002). Against this background, the title compound, (I) (Fig. 1), was synthesized and its crystal structure is reported here.



Most of the bond lengths and angles have normal values and are comparable with those reported for similar structures (Sagar *et al.*, 1999; Mahendra *et al.*, 2004). The 1,4-DHP ring has a boat conformation, with atoms N1 and C4 displaced by 0.126 (3) and 0.271 (3) Å, respectively, from the mean plane

Received 30 June 2005

Accepted 8 July 2005

Online 16 July 2005

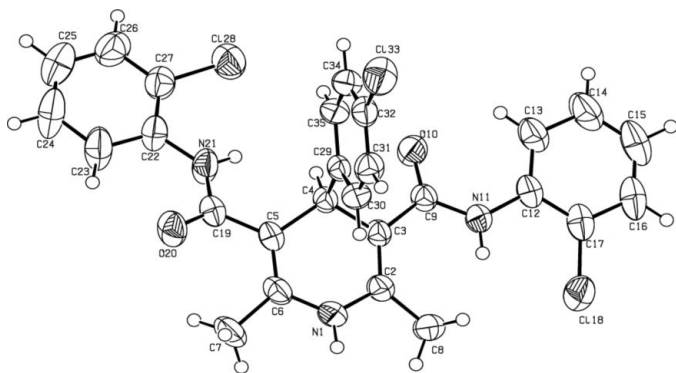


Figure 1
View of (I), shown with 50% probability displacement ellipsoids.

through the other four C atoms. The other structural characteristics are the conformations of the 2-chlorophenyl-carbamoyl groups on the dihydropyridine ring. Each carbonyl group of 2-chlorophenylcarbamoyl can be oriented in a synperiplanar (*cis*) or antiperiplanar (*trans*) conformation with respect to the adjacent C=C double bond of the dihydropyridine ring. The observed torsion angles are C2—C3—C9—O10 [168.5 (3)°] and C6—C5—C19—O20 [28.8 (5)°], indicating *trans* and *cis* conformations, respectively.

The structure exhibits both intra- and intermolecular hydrogen bonds (Table 2 and Fig. 2), which help to stabilize the crystal structure. The intermolecular hydrogen bonds N1—H1...O20ⁱ and C34—H34...O10ⁱⁱ (symmetry codes as in Table 2) link the molecules into infinite one-dimensional chains (Fig. 2).

Experimental

A mixture of 2-chloroacetoacetanilide (4.22 g, 0.02 mol) and 4-chlorobenzaldehyde (1.40 g, 0.01 mol) was heated continuously at reflux for 15 min, with stirring, using 30 ml methanol as solvent. Ammonia (4 ml) was added to the reaction mixture after 3 h. A light-yellow product separated in the flask; this was filtered off, washed twice with methanol and finally dried in a hot-air oven. Recrystallization from dimethylformamide gave a yield of 53–54% (m.p. 472 K). Analysis calculated: C 64.37, H 4.82, Cl 13.57, N 8.04, O 9.19%. 3 g was taken up in 30 ml of methanol/dimethylformamide (8:2). Charcoal (3 g) was added and the solution heated for 5 min. It was then filtered while hot through a Whatman 42 filter paper. The solution was kept for 30 d in a conical flask with the stopper left slightly open, and pale-yellow crystals grew by evaporation.

Crystal data

$C_{27}H_{22}Cl_3N_3O_2$	$D_x = 1.440 \text{ Mg m}^{-3}$
$M_r = 526.83$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 8244 reflections
$a = 20.566 (2) \text{ \AA}$	$\theta = 2.2\text{--}27.9^\circ$
$b = 10.698 (1) \text{ \AA}$	$\mu = 0.41 \text{ mm}^{-1}$
$c = 22.789 (2) \text{ \AA}$	$T = 295 (2) \text{ K}$
$\beta = 104.300 (2)^\circ$	Block, pale yellow
$V = 4858.6 (6) \text{ \AA}^3$	$0.25 \times 0.2 \times 0.2 \text{ mm}$
$Z = 8$	

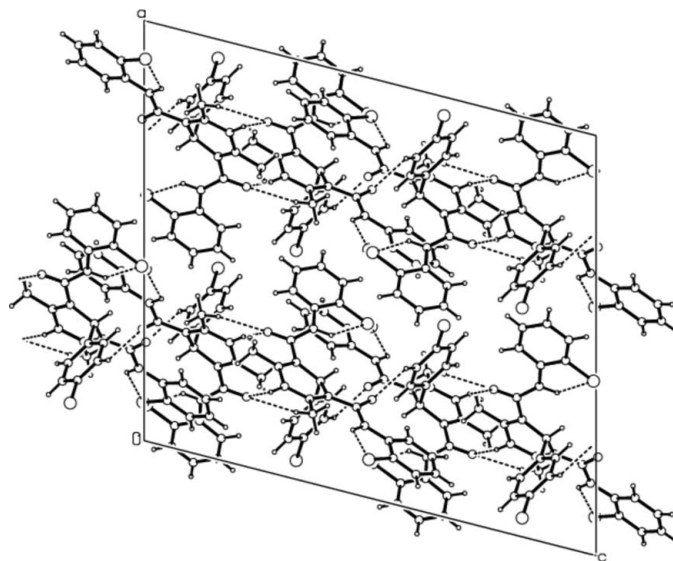


Figure 2
The crystal packing in (I), viewed down the *b* axis. Dashed lines indicate hydrogen bonds.

Data collection

MacScience DIPLabo 32001
diffractometer
 ω scans
Absorption correction: none
8244 measured reflections
4452 independent reflections

3458 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$
 $\theta_{\text{max}} = 27.9^\circ$
 $h = -23 \rightarrow 24$
 $k = -10 \rightarrow 11$
 $l = -29 \rightarrow 29$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.181$
 $S = 1.06$
4452 reflections
318 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0987P)^2 + 5.2071P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.57 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.54 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

Cl18—C17	1.740 (3)	N1—C6	1.376 (4)
Cl28—C27	1.735 (4)	N11—C9	1.357 (4)
Cl33—C32	1.739 (3)	N11—C12	1.404 (4)
O10—C9	1.213 (4)	N21—C19	1.353 (4)
O20—C19	1.227 (3)	N21—C22	1.410 (5)
N1—C2	1.382 (4)		
C2—N1—C6	124.0 (2)	Cl18—C17—C12	120.0 (3)
C9—N11—C12	129.0 (2)	Cl18—C17—C16	118.1 (3)
C19—N21—C22	128.8 (3)	O20—C19—N21	120.7 (3)
N1—C2—C3	119.1 (3)	O20—C19—C5	123.5 (3)
N1—C2—C8	112.0 (2)	N21—C19—C5	115.8 (2)
N1—C6—C5	119.5 (3)	N21—C22—C23	124.0 (3)
N1—C6—C7	112.9 (3)	N21—C22—C27	118.5 (3)
O10—C9—N11	120.8 (2)	Cl28—C27—C22	119.8 (3)
O10—C9—C3	120.2 (3)	Cl28—C27—C26	118.1 (3)
N11—C9—C3	119.1 (2)	Cl33—C32—C31	119.8 (2)
N11—C12—C17	118.3 (3)	Cl33—C32—C34	119.5 (2)
N11—C12—C13	124.2 (3)		

Table 2
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1\cdots O20^i$	0.86	2.05	2.891 (3)	167
$N11-H11\cdots Cl18$	0.86	2.46	2.943 (3)	116
$N21-H21\cdots Cl28$	0.86	2.48	2.935 (3)	114
$C4-H4\cdots N21$	0.98	2.54	2.905 (4)	102
$C7-H7C\cdots O20$	0.96	2.36	2.838 (4)	110
$C8-H8B\cdots O20^i$	0.96	2.56	3.423 (4)	149
$C13-H13\cdots O10$	0.93	2.24	2.833 (5)	121
$C23-H23\cdots O20$	0.93	2.28	2.842 (5)	119
$C34-H34\cdots O10^{ii}$	0.93	2.49	3.227 (4)	136

Symmetry codes: (i) $-x + \frac{1}{2}, +y - \frac{1}{2}, -z - \frac{1}{2}$; (ii) $-x + \frac{1}{2}, -y + \frac{5}{2}, -z$.

H atoms were placed at idealized positions and allowed to ride on their parent atoms, with C—H distances in the range 0.93–0.98 Å and N—H = 0.86 Å; $U_{iso}(H)$ values were set equal to $xU_{eq}(\text{carrier atom})$, where $x = 1.5$ for methyl H atoms and 1.2 for all other H atoms.

Data collection: *XPRESS* (MacScience, 2002); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

We thank the DST, Government of India, for financial assistance (project SP/I2/FOO/93). MM thanks the CSIR, Government of India, for the award of a Senior Research Fellowship.

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